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Micro squeeze flow rheometer for high frequency analysis of nano-litre volumes of viscoelastic fluid

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D. Cheneler ^{a*}, J. Bowen ^b, M. C. L. Ward ^a, M. J. Adams ^b

^a Dept. of Mechanical Engineering, University of Birmingham, Birmingham, B15 2TT, UK

^b Dept. of Chemical Engineering, University of Birmingham, Birmingham, B15 2TT, UK

e-mail: D.Cheneler@bham.ac.uk

ABSTRACT

In this paper, the design, fabrication and experimental analysis of a piezoelectrically actuated micro squeeze flow rheometer is presented. Being only 30x30x0.5 mm in size, the micro rheometer is sensitive to very small volumes of the order of 1-10 nano-litre of liquid and light enough to operate at frequencies in the kHz regime, an order of magnitude higher than normally attainable with conventional cone and plate rheometry. Initial experiments show that the response of the rheometer is dependent on the viscoelasticity of the fluid being tested. The prototype was used to measure the moduli of poly(dimethylsiloxane) (PDMS) of viscosity 10 Pa s, a non-volatile viscoelastic fluid, over the frequency range of 10-1000 Hz. Results show good agreement between the moduli measured using conventional rheometry up to 100 Hz and with values extrapolated up to 1 kHz.

1. Introduction

In many industries, such as the polymer and pharmaceutical industries, the rheology of fluids is of supreme importance as it directly affects a products performance [1]. Modern research into the synthesizing of new materials has lead to the development of high-throughput screening [2] and combinatorial chemistry [3]. This requires the rapid synthesis and automated experimentation on a large number of different but structurally related materials. However, conventional bulk rheometers, such as cone and plate rheometers, are still being used to measure the viscoelastic properties of materials. This is because microrheology is a recent field still undergoing rapid development [4] and so reliable, accurate and multiplexable micro rheometers are still not commercially available. This is an issue because conventional rheometers require relatively large volumes of fluid and only operate at low frequencies, typically up to 100 Hz.

The micro squeeze flow rheometer (MSFR) described in this paper has been designed to be sensitive to nano-litre volumes of liquid and operates up to 1 kHz. Its size and the method of fabrication used also makes it very cheap to produce allowing it to be operated in parallel with a large array of similar devices. This will greatly facilitate high through-put screening of large numbers of small volumes of fluid, which is highly desirable within in the chemical and pharmaceutical industries. It also makes it possible to integrate the rheometer into lab-on-a-chip devices or use it for BioMEMS applications.

This paper describes the design, fabrication and experimental analysis of the MSFR. The theory developed in [5] is applied to the data obtained from the response of the MSFR for the 10 Pa s poly(dimethylsiloxane) (PDMS) to calculate the storage and loss moduli of the fluid. The results are compared to that obtained using conventional cone and plate rheometry and show good agreement.

2. Design

The MSFR consists of two parts: an active upper layer and a passive lower layer. The active layer is comprised of a partially laminated plate whereby a piezoelectric disc is bonded onto a thin circular silicon diaphragm (see Fig. 1). At the centre of the diaphragm is a small cylindrical platen directly above a similar platen which is fixed to the lower layer. Prior to experimentation, a viscoelastic fluid sample is placed onto the lower platen. This sample takes the form of a liquid pendular bridge between the two platens when the two parts of the MSFR is assembled.

Fig. 1. Schematic of the two parts of the MSFR.

The piezoelectric disc is partially coated with concentric electrodes which allow a potential to be applied across part of the disc, but also allows the induced voltage due to the strain in the piezoelectric material to be measured. The disc is polarized so that it expands radially when a potential is applied across it. This causes the diaphragm to deflect, moving the upper platen up and down. This movement imposes a force onto the fluid thus squeezing it. The resultant induced voltage due to the strain caused by the applied voltage and resisting force of the fluid is therefore a function of the fluid properties. In an earlier paper [6], a generic compliant oscillating squeeze flow rheometer was considered. It was shown that the storage and loss modulus of a viscoelastic liquid that can be described by the generalised Maxwell model can be calculated from the response of a MEMS device characterised in terms of its mass and stiffness, provided the amplitude of the oscillations is small. This work

was expanded upon [5] and applied to the MSFR. The rheometer was analysed in terms of its electro-mechanical behaviour and it was shown how the electrical response of the device can be used to determine the mechanical response and hence the storage and loss modulus of the liquid.

3. Fabrication

The active layer was fabricated from a BSOI wafer with a conductive 50 μm thick device layer (see Fig. 2). First, the handle wafer was patterned using SPR220-7 photoresist and etched through to the oxide to define a series of thin concentric rings between the upper platen and edge of the diaphragm. The piezo discs were purchased coated with silver electrodes on both sides, one side was completely coated and one side was patterned using S1813 photoresist. The electrodes were then isolated by submerging the disc in nitric acid to dissolve the excess silver. The disc was glued on to the centre of the diaphragm and the supporting silicon rings were removed by etching the underlying oxide away by submerging the area in HF acid. The upper platen was then coated with a 250 nm layer of C_4F_8 . This was done to modify the surface energy of the platen in order to prevent the test fluid flowing away during experimentation.

Fig. 2. Schematic of the fabrication steps of the active layer of the MSFR.

The passive layer comprises of the lower platen, a sunken area to allow excess fluid to flow away and alignment features defined by etching the substrate using DRIE to three different heights as shown in Fig. 1. It is made from a 525 μm thick silicon wafer. In each step the substrate was first patterned using SPR 220-7 photoresist, before being etched to the required depth. As before, the lower platen was coated with a layer of C_4F_8 . For assembly, before experimentation, a liquid drop is placed on the lower platen and the upper part of the rheometer is brought down into contact with the lower part and clamped together.

4. Experimentation

The MSFR was excited by applying a sinusoidal voltage with constant amplitude of 1 V and frequency of 10-1000 Hz using a function generator by placing probes onto the active electrodes on the piezoelectric material on the rheometer. The passive electrode was connected via a separate probe to a unity gain buffer amplifier, to

negate impedance effects, to an ADC-212 Picoscope. The ground for both the active and passive electrodes is common and connected to the silicon diaphragm, which is conductive. The input voltage and induced voltage were recorded simultaneously by the Picoscope. The data was then analysed to find the amplitude of the induced voltage and phase difference between the input and induced voltage as a function of frequency. The fluids tested were PDMS with zero shear rate viscosities of 5, 10, 30, 65, 100 and 340 Pa s, used because PDMS has a low volatility. The liquids are introduced into the MSFR by placing a drop of liquid on the lower platen with a glass rod prior to assembly. The platens are easily cleaned with a pertinent solvent, in this case toluene, between tests of different fluids. In Fig. 3 it is clearly seen that the response of the MSFR is dependent on the viscoelasticity of the fluid sample in that the phase and amplitude of the response changes in relation to the viscosity of the test fluid. Note there is a complex interplay between the phase, amplitude and frequency of the induced voltage on the calculation of the elastic and viscous properties of the fluid. It is also important to note that this interplay is also temperature dependent. This can be taken advantage of by using the time-temperature superposition principle to extend the apparent frequency of the MSFR as the device is easily cooled. However, care needs to be taken as not only are the liquids viscoelastic properties temperature dependent but so are the material properties of the MSFR. Differences in the thermal expansion coefficients of the piezoelectric and silicon layers can lead to excess stress in the device which may manifest itself as an apparent shift in the elastic properties of the liquid.

Fig. 3. The effect of fluid viscoelasticity on the induced voltage for a range of frequencies. The amplitude (a) and phase (b) is plotted against the zero shear rate of the viscosity. ♦ denotes 10 Hz response; ■ denotes 100 Hz response and ▲ denotes 500Hz response.

In order to check the accuracy of the microrheometer it was necessary to compare the results to those obtained by conventional oscillatory rheometry. The storage and loss moduli of PDMS of viscosity 10 Pa s, was measured from 10-100 Hz using an AR2000 cone and plate rheometer with a 20 mm stainless steel cone with a 4° side wall angle. The amplitude of the strain was constant at 1 %. The sample volume was approximately 0.15 mL and the sample temperature was maintained at 25 °C. For this liquid, a good fit was obtained by assuming a single element Maxwell model by using a least-square method of curve fitting [5]. The pertinent viscous and elastic coefficients can be shown to be $\eta = 9.773$ Pa s, $G = 21138.8$ Pa respectively, which gave a relaxation time $\lambda = \eta/G = 0.00046$ s. These coefficients were used to calculate the moduli at higher frequencies for

comparison with results from the MSFR. Figs. 4 and 5 show that the data obtained from the MSFR are in good agreement with the measured cone and plate data for the low frequency regime, and in reasonable agreement with the values extrapolated from the Maxwell model for the high frequency regime. This suggests the MSFR behaves as suggested in [5] and is therefore a potentially useful tool for measuring high frequency properties of nano-litre volumes of viscoelastic fluids.

Fig. 4. Storage moduli for 10 Pa s PDMS obtained from cone and plate rheometry, the MSFR and extrapolated data.

Fig. 5. Loss moduli for 10 Pa s PDMS obtained from cone and plate rheometry, the MSFR and extrapolated data.

5. Conclusions

In this paper a design for a MSFR has been presented. It has been shown that the MSFR can analyse a sample size of the order of nanolitres which is a much smaller volume of fluid than cone and plate rheometry requires. It also extends the frequency range at which fluids can be tested an order of magnitude beyond the capabilities of cone and plate rheometry. It has been shown that experiments, which when combined with the theory presented in [5], produces data which is comparable with conventional cone and plate rheological data.

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